



UNIVERSITI PUTRA MALAYSIA

**SYNTHESIS, CHARACTERIZATION AND APPLICATION OF
POLYACRYLAMIDE GRAFTED SAGO STRACH FOR
COAGULATION AND FLOCCULATION PROCESSES**

ISAM YASSIN M. QUDSIEH.

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**DOCTOR OF PHILOSOPHY
UNIVERSITI PUTRA MALAYSIA**

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By

ISAM YASSIN M. QUDSIEH

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia
in Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

January 2006



DEDICATION

*Especially Dedicated to My Beloved
Mother... Father, Dr. Yassin... Brothers... Sisters...
My Wife Jomana... and to My Daughter Shams
Who always supported me in many ways...*

Abstract of thesis presented to the Senate of Universiti Putra
Malaysia in fulfilment of the requirement for the degree Doctor of
Philosophy

**SYNTHESIS, CHARACTERIZATION AND APPLICATION OF
POLYACRYLAMIDE GRAFTED SAGO STARCH FOR COAGULATION
AND FLOCCULATION PROCESSES**

By

ISAM YASSIN M. QUDSIEH

June 2005

**Chairman: Associate Professor Fakhru'l-Razi Ahmadun,
PhD**

Faculty: Engineering

Graft copolymerization of polyacrylamide (PAm) onto sago starch (S.S), S.S-g-PAm1, S.S-g-PAm2, S.S-g-PAm3, and S.S-g-PAm4, were carried out by a free radical initiation using ceric ion (CAN)-induced redox polymerization technique at $30 \pm 1^\circ\text{C}$, and under nitrogen gas atmosphere by varying the amount of CAN and PAm. The copolymers produced were characterized by Fourier Transform Infrared Spectrophotometry (FT-IR), Thermogravimetric analysis (TGA), elemental and gravimetric analyses. The FT-IR spectra of the copolymers clearly indicated the presence of characteristic peaks of PAm and sago starch, which suggested that PAm had been successfully grafted onto the sago starch. TGA thermograms showed that the copolymerization products were made up of two different

polymers. The copolymers of various percentages of yield and conversion were hydrolyzed with 0.5 M hydrochloric acid and the viscosity measurements of PAm produced from the copolymers were carried out using Ubbelohde viscometer. The average molecular weight of the side chains (M_v) of the copolymers were 5.6×10^5 , 3.1×10^5 , 7.4×10^5 , and 2.0×10^5 respectively and viscosity values were 251.97, 157.52, 314.43 and 110.02 mL/g respectively.

A series of standard jar tests were conducted in order to evaluate the performance of the polymers prepared (S.S-g-PAm) in kaolin suspension. Among the graft copolymers, S.S-g-PAm1 was selected based on the performance and sago starch content to be used in further experiments; further more, a series of standard jar tests were conducted to optimize the operation conditions of S.S-g-PAm1 in bringing down the turbidity of supernatant liquid of 200 NTU kaolin suspension by varying the following parameters: dosage, pH, speed of rapid mixing, time of rapid mixing, speed of low mixing, and time of low mixing. And the optimum conditions were found to be 0.5 mg/L, 6.3, 75 rpm, 4 minutes, 25 rpm, 5 minutes respectively. Substantial reduction of residual turbidity was achieved by S.S-g-PAm.

Settling column analysis was established for S.S-g-PAm1 in low, medium to high (50, 200, 300 NTU, respectively) initial kaolin

suspension turbidity. Initial turbidity and settling time were found to be one of the major factors in settling water coagulated with S.S-g-PAm1. Residual turbidity was found to decrease with increasing settling time; the decrease was higher for kaolin water with higher initial turbidities.

Four river samples were collected from two locations; two samples of low and medium to high turbidity (30.5, 780 NTU), respectively were collected from Hulu Langat Water Treatment Plant, and another two samples of the same level of turbidity (low and medium to high turbidity) (68 and 450 NTU), respectively were collected from Semenyih Water Treatment Plant. Samples of low to high turbidities from two different rivers in Malaysia were collected and compared with the existing coagulant (liquid alum) that is being widely used by the treatment plants. The performance of S.S-g-PAm1 were found to be good as compared to liquid alum with the advantages of the biodegradability of S.S-g-PAm1 as well as less dosage of S.S-g-PAm1 used as compared to liquid alum.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**SINTESIS, PENCIRIAN DAN PENGGUNAAN KANJI SAGU
TERCANTUM POLIAKRALAMIDA UNTUK PROSES KOAGULASI
DAN FLOKULASI**

Oleh

ISAM YASSIN M. QUDSIEH

June 2005

Pengerusi: Profesor Madya Fakhru'l-Razi Ahmadun, PhD

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Pengkopolimeran cantum poliakralamida (PAm) ke atas kanji sagu (S.S), S.S-g-PAm1, S.S-g-PAm2, S.S-g-PAm3 dan S.S-g-PAm4 telah dijalankan secara rangsangan radikal bebas menggunakan teknik polimerisasi redoks menggunakan serum ammonium nitrat (SAN) pada suhu $30 \pm 1^\circ\text{C}$, dan di dalam atmosfera gas nitrogen dengan mengubah amaun SAN dan PAm. Ko-polimer yang telah dihasilkan telah dicirikan dengan spektrofotometer inframerah (FT-IR), analisis termogravimetri (TGA), elemental dan analisis secara gravimetrik. Spektra FT-IR ko-polimer jelas menunjukkan kehadiran puncak kumpulan berfungsi PAm dan kanji sagu yang mana ini mencadangkan bahawa PAm telah dicantum dengan jayanya ke atas kanji sagu. Termogram TGA menunjukkan bahawa produk ko-

polimerisasi telah dihasilkan dari dua polimer yang berbeza. Kopolimer yang berbeza dari segi peratusan hasil dan penukaran telah dihidrolisasikan dengan 0.5 M asid hidroklorik dan pengukuran kelikatan larutan PAm telah dilakukan dengan menggunakan Ubbelohde viscometer. Jisim molekul purata kelikatan (M_v) kopolimer berkenaan masing-masing adalah 5.6×10^5 , 3.1×10^5 , 7.4×10^5 dan 2.0×10^5 dan nilai kelikatan masing-masing adalah 251.97, 157.52, 314.43, dan 110.02 mL/g.

Biodegradasi Sintetik ke atas kanji (S.S-g-PAm) berasaskan kanji sagu telah direkabentuk sebagai koagulan kationik bagi mengurangkan kekeruhan dalam air keruh tiruan (larutan rendaman kaolin) dan juga air sungai. Satu siri ujian balang piawai telah dilakukan bagi menilai prestasi polimer dalam larutan rendaman kaolin. Di antara kesemua kopolimer cantum, S.S-g-PAm1 telah dipilih berdasarkan kepada prestasinya dan kandungan kanji sagu yang akan digunakan dalam pengkajian selanjutnya. Selain daripada itu, beberapa siri ujian balang telah dijalankan untuk mengoptimumkan keadaan tindak balas S.S-g-PAm1 dalam mengurangkan kekeruhan larutan rendaman kaolin (200 NTU) dengan mengubah parameter-parameter berikut dan keadaan tindak balas optimum yang telah diperolehi adalah seperti berikut: dosej; 0.5 mg/L, pH; 6.3, kelajuan pencampuran secara laju; 75 rpm, masa

pencampuran secara laju; 4 minit, kelajuan pencampuran secara perlahan; 25 rpm dan masa pencampuran secara perlahan 5 minit. Dengan menggunakan S.S-g-PAM didapati dengan jelasnya kekurangan dalam kekeruhan larutan telah dicapai.

Analisis pemendakan telah dilakukan ke atas S.S-g-PAM1 bagi kekeruhan larutan rendaman kaolin yang rendah, sederhana dan tinggi (50, 200, 300 NTU). Didapati bahawa kekeruhan awal dan tempoh pemendakan adalah faktor utama yang mempengaruhi dalam pengkogulasian air dengan S.S-g-PAM1. Didapati juga bahawa kekurangan dalam kekeruhan larutan berkurang dengan pertambahan tempoh pemendakan. Kekurangan berkenaan lebih besar bagi air kaolin yang mempunyai kekeruhan awal yang tinggi.

Empat sampel telah diambil dari dua lokasi; dua sampel dengan masing-masing kekeruhan rendah dan kekeruhan sederhana ke kekeruhan tinggi (30.5, 780 NTU) telah diperolehi dari Loji Rawatan Air Hulu Langat dan dua sampel lain dengan tahap kekeruhan yang sama (rendah dan sederhana ke tinggi) (68 dan 450 NTU) telah diperolehi dari Loji Rawatan Air Semenyih. Sampel air bagi kekeruhan rendah ke kekeruhan tinggi dari sungai-sungai di Malaysia telah diperolehi dan perbandingan telah dibuat dengan koagulan yang sediaada iaitu alum cecair yang digunakan secara

meluas di loji-loji perawatan air. Didapati hasil dari prestasi S.S-g-PAm1 adalah baik jika dibandingkan dengan alum cecair dengan kelebihan S.S-g-PAm1 yang boleh berbiodegradasi dan dosejnya adalah kurang berbanding cecair alum.

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I certify that an Examination Committee has met on 5th of January 2006 to conduct the final examination of Isam Yassin M. Qudsieh on his Doctor of Philosophy thesis entitled "Synthesis, Characterization and Application of Polyacrylamide Grafted Sago Starch For Coagulation and Flocculation Processes" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

I-Y. Qudsieh


ISAM YASSIN M. QUDSIEH

Date: 20/3/2006

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LIST OF SYMBOLS

PAm	Polyacrylamide
CAN	Cerric Ammonium Nitrate
PPS	Potassium Peroxy disulphate
APS	Ammonium Persulphate
S.S	Sago Starch
S.S-g-PAm	Polyacrylamide Grafted Sago Starch
$[\eta_{sp}]$	Specific Viscosity
$[\eta_{red}]$	Reduced Viscosity
M_v	Side Chains Average Molecular Weight
S.S-g-PAm1	Polyacrylamide Grafted Sago Starch 1
S.S-g-PAm2	Polyacrylamide Grafted Sago Starch 2
S.S-g-PAm3	Polyacrylamide Grafted Sago Starch 3
S.S-g-PAm4	Polyacrylamide Grafted Sago Starch 4
S.S-g-PAm(1-4)	Polyacrylamide Grafted Sago Starch 1, 2,3, and 4
AGU	Anhydroglucose Unit
FT-IR	Frontier Transform Infra Red
TGA	Thermogravimetric Analysis
NTU	Nephelometric Turbidity Unit